in the molecules labelled *a* in Table 1 a methyl hydrogen is almost coplanar with the nonhydrogen atoms, but perpendicular to them in the molecules labelled *b*. The average torsion angles (modulo 60°) over the C(1)–C(2) bond and relative to any of the nitrogen atoms are 6 (9) and 28 (9)°, respectively. The methyl rotational barrier for this type of compounds is very low. Different types of calculations indicate a magnitude of a few kJ mol^{-1} and the methyl-group orientation is most probably determined by intermolecular interactions. For further discussions on this subject on similar molecules, see *e.g.* Hagler, Leiserowitz & Tuval (1976); Jeffrey, Ruble, McMullan, DeFrees, Binkley & Pople (1980); Norrestam *et al.* (1983).

The molecular packing in the crystal structure is shown in Fig. 2. The structure is held together by *inter alia* an extensive net of intermolecular hydrogen bonds. The four symmetry-independent amino groups are hydrogen donors to two hydrogen bonds each, while the oxygen atoms of the carbonate ions are acceptors of three hydrogen bonds each. The N···O distances range from 2·757 (5) to 2·864 (5) Å. The water molecule at O(*w*) participates (as hydrogen donor) in only one hydrogen bond, 2·830 (4) Å, to one of the carbonate oxygen atoms, O (1). Thus, the water molecule is rather loosely bonded in the structure. This is also reflected by the significantly higher thermal vibration parameter of O(*w*), $U_{\text{eq}} = 0{\cdot}090$ (2) Å^2, compared with the average value 0·018 (1) Å^2 of the other atoms.

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Thujaplicatin Methyl Ether, $C_{21}H_{24}O_7$

By Mino R. Caira

University of Port Elizabeth, Port Elizabeth, South Africa

William H. Watson*

FASTBIOS Laboratory, Department of Chemistry, Texas Christian University, Forth Worth, TX 76129, USA

and Mario Silva

Universidad de Concepción, Concepción, Chile

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Abstract. $M_r = 388{\cdot}4$, monoclinic, $P2_1$, $a = 11{\cdot}715$ (2), $b = 10{\cdot}815$ (1), $c = 7{\cdot}411$ (1) Å, $\beta = 92{\cdot}66$ (1)°, $V = 983{\cdot}0$ (2) Å^3, $Z = 2$, $D_m = 1{\cdot}365$ (5), $D_x = 1{\cdot}375$ Mg m^{-3}, $\lambda(\text{Cu } K\alpha) = 1{\cdot}5418$ Å, $\mu = 0{\cdot}87$ mm^{-1}, $F(000) = 412{\cdot}0$, $T = 298$ K, $R = 0{\cdot}033$ for 1314 unique reflections. The lactone ring has a twist conformation [torsion angle C–C–C–O 27·6 (3)°] and the substituent carbon atoms at the 2- and 3-positions are *trans* to one another. Intermolecular O–H···O hydrogen bonding involving both the carbonyl and phenolic groups stabilizes the crystal structure. The absolute configuration assigned previously by circular dichroism studies has been confirmed.

Introduction. *Podocarpus saligna* D. Don is one of three species of *Podocarpus* native to Chile. The constituents daucosterin, fern-9(11)-ene, isofernene, β-sitosterol and *n*-nonacosane (Bhakuni, Bittner, Sammes & Silva, 1974) as well as more than ten nor- and bisnorditerpene dilactones (Zabel, Watson, Silva,

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* To whom correspondence should be addressed.

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Bittner & Langs, 1980; Watson, Zabel, Silva & Bittner, 1982) have been isolated from the roots and leaves of *P. saligna*. The nor- and bisnorditerpene dilactones from *Podocarpus* species exhibit a variety of biological activities, and chemical investigations of these species are still in progress. The compound thujaplicatin methyl ether was isolated from the roots of *P. saligna*. The same compound has been isolated from *Thuja standishii* and the absolute configuration assigned by circular dichroism (CD) studies (Nishibe, Hisada & Inagaki, 1974). The structure and confirmation of absolute configuration are reported.

Experimental. Details of data collection and least-squares refinement given in Table 1. All but one of the non-hydrogen atoms located in E map phased by *MULTAN*78 (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978); remaining C atom and most H atoms revealed in subsequent difference syntheses, but the latter included at geometrically constrained positions (C—H, O—H = 1·08 Å) with U_{iso} held at 1·5 times those of parent atoms; anisotropic temperature factors assigned to all non-hydrogen atoms in final cycles of refinement; all calculations performed with XRAY76 (Stewart, Machin, Dickinson, Ammon, Heck & Flack, 1976); anomalous dispersion correction for O scattering factors (*International Tables for X-ray Crystallography*, 1962) applied to distinguish absolute configuration by the R-factor ratio test (Hamilton, 1965).*

Table 1. *Experimental and refinement parameters*

Density measurement	Flotation in aq. KI
Crystal dimensions (mm)	0·43 × 0·43 × 0·31
Diffractometer system	Syntex $P2_1$
Lattice parameters	Least-squares refinement of angular data for 15 reflections (6·0 < θ < 14·1°)
$(\sin\theta/\lambda)_{\max}$ for intensity measurements (Å^{-1})	0·562
$(h\ k\ l)_{\min}$	0 0 −8
$(h\ k\ l)_{\max}$	12 11 8
Scan mode	θ:2θ
Scan speed	Variable: range 3·91–29·30° min^{-1}
Indices of standard reflection	0 0 1
Stability of standard reflection	±0·7%
Absorption corrections	None
Number of reflections measured	1478
Number of unique reflections	1356
Number of observed reflections [$I > 3\sigma(I)$]	1314
Quantity minimized	$\sum w(\lvert F_o\rvert - \lvert F_c\rvert)^2$, $w = 1/\sigma^2(F_o)$
Number of parameters refined	252
R	0·033
wR	0·047
S	2·944
Average Δ/σ	0·12
Maximum Δ/σ	1·04
Constrained U_{iso} (H)	Range: 0·063–0·104 Å^2
Highest $\Delta\rho$ excursion in difference map	0·17 e Å^{-3}

Table 2. *Fractional atomic coordinates* (×10^4) *for non-hydrogen atoms and equivalent isotropic thermal parameters* U_{eq} (Å^2× 10^3) *with e.s.d.'s in parentheses*

	x	y	z	U_{eq}*
C(1)	8063 (3)	4265	4783 (5)	52 (2)
O(1)	7745 (2)	5274 (2)	4353 (4)	65 (1)
C(2)	8411 (2)	3209 (3)	3558 (4)	43 (2)
C(3)	8531 (2)	2103 (3)	4838 (4)	42 (2)
C(4)	8720 (3)	2721 (4)	6666 (4)	53 (2)
O(5)	8156 (2)	3920 (3)	6513 (3)	61 (1)
C(6)	7603 (2)	3075 (3)	1869 (4)	45 (2)
C(7)	6366 (2)	2850 (3)	2257 (4)	41 (1)
C(8)	5652 (2)	3849 (3)	2528 (4)	43 (2)
C(9)	4524 (2)	3673 (3)	2955 (4)	42 (2)
C(10)	4099 (2)	2482 (3)	3140 (4)	44 (2)
C(11)	4794 (3)	1488 (3)	2807 (4)	47 (2)
C(12)	5926 (2)	1660 (3)	2360 (4)	44 (2)
O(9)	3764 (2)	4608 (2)	3250 (3)	53 (1)
O(10)	2997 (2)	2338 (2)	3624 (3)	55 (1)
O(11)	4265 (2)	372 (3)	2971 (4)	70 (2)
C(13)	4083 (3)	5810 (4)	2681 (5)	63 (2)
C(14)	4853 (4)	−723 (4)	2491 (5)	67 (2)
C(15)	9495 (3)	1195 (3)	4466 (4)	49 (2)
C(16)	9343 (2)	557 (3)	2665 (4)	44 (2)
C(17)	8498 (3)	−330 (3)	2377 (4)	49 (2)
C(18)	8305 (2)	−865 (3)	693 (4)	45 (2)
C(19)	8980 (2)	−543 (3)	−725 (4)	43 (2)
O(18)	7442 (2)	−1678 (3)	276 (3)	70 (2)
O(19)	8816 (2)	−1060 (2)	−2411 (3)	52 (1)
C(20)	9848 (3)	306 (3)	−433 (4)	49 (2)
C(21)	10024 (3)	855 (3)	1248 (4)	48 (2)
C(22)	7291 (3)	−2684 (4)	1461 (6)	72 (2)

* $U_{\text{eq}} = \frac{1}{3}[U_{11}(aa^*)^2 + U_{22}(bb^*)^2 + U_{33}(cc^*)^2 + 2U_{13}aca^*c^*\cos\beta + 2U_{23}bcb^*c^*\cos\alpha + 2U_{12}a^*b^*ab\cos\gamma]$.

Table 3. *Interatomic distances* (Å) *and principal torsion angles* (°) *with e.s.d.'s in parentheses*

C(1)—O(1)	1·192 (3)	C(10)—O(10)	1·365 (3)
C(1)—C(2)	1·526 (4)	C(11)—O(11)	1·365 (5)
C(2)—C(3)	1·529 (4)	C(13)—O(9)	1·422 (5)
C(3)—C(4)	1·518 (4)	C(14)—O(11)	1·423 (5)
C(4)—O(5)	1·457 (5)	C(3)—C(15)	1·531 (4)
O(5)—C(1)	1·335 (4)	C(15)—C(16)	1·506 (4)
C(2)—C(6)	1·541 (4)	C(16)—C(17)	1·388 (4)
C(6)—C(7)	1·510 (3)	C(17)—C(18)	1·385 (4)
C(7)—C(8)	1·387 (4)	C(18)—C(19)	1·389 (4)
C(8)—C(9)	1·386 (4)	C(19)—C(20)	1·380 (4)
C(9)—C(10)	1·390 (4)	C(20)—C(21)	1·387 (4)
C(10)—C(11)	1·378 (5)	C(21)—C(16)	1·386 (4)
C(11)—C(12)	1·394 (4)	C(18)—O(18)	1·364 (4)
C(12)—C(7)	1·390 (4)	C(19)—O(19)	1·374 (4)
C(9)—O(9)	1·372 (4)	C(22)—O(18)	1·414 (5)

C(1)—C(2)—C(6)—C(7)	−58·5 (3)	C(3)—C(15)—C(16)—C(17)	−71·1 (4)
C(2)—C(6)—C(7)—C(8)	86·7 (3)	C(6)—C(2)—C(3)—C(15)	91·5 (3)
C(2)—C(3)—C(15)—C(16)	−63·5 (3)		

Discussion. Table 2 lists final atomic coordinates. Bond lengths and principal torsion angles are listed in Table 3.

Fig. 1 is a perspective view of the molecule showing the thermal ellipsoids of the non-hydrogen atoms and the numbering scheme. All data refer to the 2R,3R absolute configuration. The ratio $wR(2)/wR(1)$ (1 = 2R,3R, 2 = 2S,3S) is 1·02 while $\mathscr{R}_{1,1062,0\cdot005} = 1\cdot004$ (Hamilton, 1965), indicating that the 2S,3S configuration can be rejected at the 0·5% level. This is

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* Lists of hydrogen-atom coordinates, anisotropic thermal parameters, valence angles, selected torsion angles, equations of least-squares planes and structure factors have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 38937 (17 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

consistent with the CD assignment of the $2R,3R$ configuration to thujaplicatin methyl ether isolated from *Thuja standishii* (Gord.) Carr. (Nishibe, Hisada & Inagaki, 1974). The overall molecular conformation is best described in terms of the principal torsion angles (Table 3). Bond lengths and valence angles in the lactone ring are similar to those reported elsewhere (Conde, Moreno & Márquez, 1980). The ring displays a twist conformation. Atoms O(1), C(1), C(2) and O(5) are coplanar [max. deviation from the least-squares plane 0·0004 Å] while atoms C(3) and C(4) are respectively 0·24 and 0·20 Å above and below the plane as viewed in Fig. 1 (uncertainties in the atomic positions at the 5% level average 0·01 Å). A *trans* relationship between the substituents at C(2) and C(3) is evident from the torsion angle C(6)—C(2)—C(3)—C(15), the deviation from 120° being due to the ring pucker. Mean-plane calculations show that the hydroxydimethoxybenzyl moiety has symmetry approximating C_{2v}. The carbonyl O atom and the two phenolic O atoms participate in two crystallographically distinct hydrogen bonds: $\mathrm{C(1)=O(1)\cdots H(10^{i})-O(10^{i})}$ with O···O 2·847 (3), O···H 1·853 (3) Å, and $\mathrm{O(10)\cdots H(19^{i})-O(19^{i})}$ with O···O 2·854 (3), O···H 1·860 (2)Å [(i) = $1-x$, $\frac{1}{2}+y$, $1-z$]. Each molecule is thus linked to 2_1-related molecules by four hydrogen bonds, atom O(10) acting as both donor and acceptor.

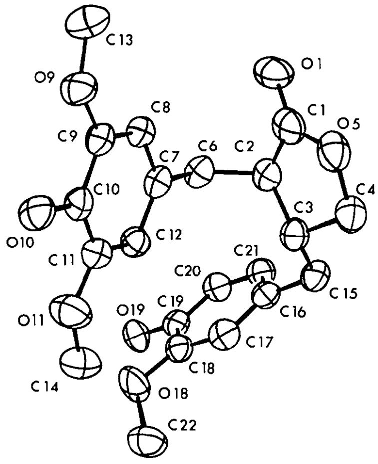

Fig. 1. *ORTEP* plot (Johnson, 1971) of the title molecule. Thermal ellipsoids are drawn at the 50% probability level. Hydrogen atoms have been omitted for clarity.

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Structure of 1*H*-Azepine-2,5-dione 5-(*O*-Methyloxime), $C_7H_8N_2O_2$

By Don T. Cromer, Robert R. Ryan and D. Scott Wilbur

University of California, Los Alamos National Laboratory, PO Box 1663, *MS C*346, *Los Alamos, New Mexico* 87545, *USA*

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Abstract. $M_r = 152{\cdot}15$, triclinic, $P\bar{1}$, $a = 3{\cdot}877$ (2), $b = 9{\cdot}522$ (2), $c = 10{\cdot}674$ (2) Å, $\alpha = 107{\cdot}99$ (2), $\beta = 97{\cdot}66$ (3), $\gamma = 98{\cdot}50$ (3)°, $V = 363{\cdot}9$ Å^3, $Z = 2$, $D_x = 1{\cdot}388$ Mg m^{-3}, Mo $K\alpha$ ($\lambda\alpha_1 = 0{\cdot}70926$ Å), $\mu = 0{\cdot}0975$ mm^{-1}, $F(000) = 160$, $T \sim 293$ K, $R = 0{\cdot}030$, 825 unique observed reflections. Compound synthesized in this laboratory. The seven-membered ring is virtually planar, although the bond lengths indicate little

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